

DEVELOP METHODS FOR PREPARATION OF PURE COPPER
(II) FLUORIDE AND DEVELOP ANALYTICAL TECHNIQUES
FOR DETERMINATION OF IMPURITIES

by
J. R. Lundquist

CASE FILE
COPY
Dupe of 43602

FACILITY FORM 502	(ACCESSION NUMBER)	(THRU)
	19	1
	(PAGES)	(CODE)
	NASA-CR-72571	(CATEGORY)
	(NASA CR OR TMX OR AD NUMBER)	

BATTELLE-NORTHWEST
prepared for
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Lewis Research Center
Contract NAS 3-10942
R. B. King, Project Manager



NOTICE

This report was prepared as an account of Government-sponsored work. Neither the United States, nor the National Aeronautics and Space Administration (NASA), nor any person acting on behalf of NASA:

- A.) Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately-owned rights; or
- B.) Assumes any liabilities with respect to the use of, or for damages resulting from the use of, any information, apparatus, method or process disclosed in this report.

As used above, "person acting on behalf of NASA" includes any employee or contractor of NASA, or employee of such contractor, to the extent that such employee or contractor of NASA or employee of such contractor prepares, disseminates, or provides access to any information pursuant to his employment or contract with NASA, or his employment with such contractor.

Requests for copies of this report should be referred to

National Aeronautics and Space Administration
Scientific and Technical Information Facility
P.O. Box 33
College Park, Md. 20740

FINAL REPORT

DEVELOP METHODS FOR PREPARATION OF PURE COPPER (II) FLUORIDE AND
DEVELOP ANALYTICAL TECHNIQUES FOR DETERMINATION OF IMPURITIES

by

J. R. Lundquist

PACIFIC NORTHWEST LABORATORIES
a division of
BATTELLE MEMORIAL INSTITUTE
3000 Stevens Drive
Richland, Washington 99352

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

June 12, 1969

CONTRACT NAS 3-10942

NASA Lewis Research Center
Cleveland, Ohio

R. B. King, Project Manager

ABSTRACT

This report describes the following methods for the preparation of ultra pure copper (II) fluoride: 1) Fluorination of copper (II) hydroxyfluoride; 2) Decomposition of a copper (II) ammonium fluoride double salt; and 3) Fluorination of copper (II) sulfide. Method 1 was used to prepare one kilogram of ultra pure copper (II) fluoride. Analytical techniques used to assure product purity are described.

SUMMARY

This program was undertaken to develop analytical techniques for the characterization of ultra pure copper (II) fluoride and also to develop methods for producing one kilogram of ultra pure copper (II) fluoride. Spark source mass spectroscopy, emission spectroscopy and neutron activation analysis was used to assure product purity. Spark source mass spectroscopy was the primary analytical tool to follow product purity during the different preparative steps. In addition to analyzing for the metallic elements it was very useful for the determination of hydrogen, oxygen, nitrogen and carbon. These elements are difficult to analyze in the presence of copper (II) fluoride, thus spark source mass spectroscopy was the only technique available for analysis of these elements.

Three different techniques were considered for making the copper (II) fluoride: fluorination of copper (II) sulfide, fluorination of copper (II) hydroxyfluoride and decomposition of a copper (II) ammonium fluoride double salt. The fluorination of the copper (II) sulfide was the least successful of the methods. The fluorination of the copper (II) hydroxyfluoride and the decomposition of the copper (II) ammonium fluoride double salt were quite successful, and either method would be acceptable for the preparation of one kilogram of ultra pure copper (II) fluoride. The fluorination of copper (II) hydroxyfluoride was chosen, as this material could be obtained in high-purity on the open market and only one step would be required to produce the final copper (II) fluoride. One kilogram of copper (II) fluoride was prepared in this manner. The final product contained less than 50 ppm by weight of any impurity element with the possible exception of magnesium, which was analyzed at 75 ppm by emission spectroscopy. Even including the highest magnesium values the over-all purity of the copper (II) fluoride is 99.98%.

INTRODUCTION

The work described herein was sponsored by the National Aeronautics and Space Administration, Lewis Research Center, and represents work directed toward development of a process for the production of ultra pure copper (II) fluoride. Copper (II) fluoride is used as a cathode material in experimental high-energy-density batteries. Studies are underway to characterize these batteries, but progress has been hindered by the general unavailability of ultra pure copper (II) fluoride in the quantities required.

Battelle-Northwest agreed to undertake a program which would lead to the preparation of one kilogram of ultra pure copper (II) fluoride. This program was to consist of a preliminary study of several different production methods and finally preparing the kilogram of copper (II) fluoride from the most promising series of chemical reactions. The reactions of interest are the preparation of copper (II) sulfide followed by fluorination, the preparation of copper (II) ammonium fluoride and subsequent decomposition under fluorine, and the fluorination of copper (II) hydroxyfluoride. Preliminary experiments showed that the fluorination of the copper (II) hydroxyfluoride would provide the easiest procedure to prepare a kilogram of material.

Chemical analysis of the various materials was done mainly by spark source mass spectrometry, emission spectroscopy and neutron activation analysis. A standard copper fluoride sample was submitted by the Lewis Research Center to cross check these three different analytical techniques. In general, the above three methods checked each other well and also agreed with the values obtained from the Lewis Research Center.

This program was designed to prepare 1 kilogram of copper (II) fluoride containing less than 50 ppm of any element and to package the final product in quartz containers under an inert atmosphere.

EXPERIMENTAL

A fluorine facility, shown in Figure I, was used in all of the fluorination experiments. Nickel, monel and copper were used as construction materials as these materials are known to become passive in the presence of fluorine, even at elevated temperatures. A nickel reaction vessel with a copper liner was fabricated to prepare large quantities of copper (II) fluoride. A schematic diagram of this reaction vessel is shown in Figure II. The shelves supporting the solid reactants were made of copper to minimize contamination of the copper (II) fluoride product. Pure nitrogen gas (99.997%) was used for purging the reaction vessel prior to placing the vessel into the inert atmosphere box, and also for diluting the fluorine gas. A dry box containing pure nitrogen was set up for handling the copper (II) fluoride after preparation. An oxygen monitor (Westinghouse Oxygen Analyzer, Model 207E) and a moisture monitor (Consolidated Electrodynamics Corporation Model 26-303) were placed in the exit stream of this system. We were able to keep the oxygen concentration below 100 ppm and the moisture content below 10 ppm. This atmosphere monitoring assured that the copper (II) fluoride would be packaged in an inert environment.

Matheson 98% fluorine was used for the fluorination step. The major impurities in this gas are hydrogen fluoride and oxygen. The oxygen concentration was not great enough to adversely affect the driving force of the chemical reactions in question. The hydrogen fluoride was removed by passing the fluorine through a sodium fluoride bed. A nickel filter was placed on the exit end of the sodium fluoride bed to prevent any particulate material from reaching the reaction vessel.

In all cases the chemicals used were of the highest purity available on the open market. Below is a list of chemicals used in this program.

Hydrochloric Acid	- Reagent grade
Nitric Acid	- Reagent grade
Water	- Triple distilled
Fluorine	- Matheson 98%. Principal impurities are hydrogen fluoride and oxygen
Hydrogen Sulfide	- Matheson 99.6%
Ammonium Fluoride	- Reagent grade. Only copper present in significant amounts, which did not interfere with the program
Copper	- 99.999%
Copper Hydroxyfluoride	- Ledoux, reagent grade

The utmost care was taken in handling these materials. High purity quartz was used to carry out reactions at elevated temperatures, and polyethylene containers were used to handle materials at room temperatures. A copper mortar and pestle was used to grind the copper (II) fluoride to a fine powder. Copper spatulas were fabricated for transferring materials and a copper funnel was used to load the final copper (II) fluoride product into the quartz containers.

ANALYTICAL FACILITIES

The purity of the copper (II) fluoride was determined by several techniques, including spark source mass spectroscopy, neutron activation analysis, and emission spectroscopy. These techniques will be described below.

Mass Spectroscopy

The mass spectrometer used to analyze the copper (II) fluoride is a Model JMS-01-B instrument manufactured by Japan Electron Optics Laboratory Company (JEOL). This instrument is a spark source, double focusing mass spectrometer having high sensitivity and high resolution for impurities in inorganic compounds. Most impurities can be easily detected at 10 ppm by weight or less. It is necessary to mix the copper (II) fluoride with high purity gold or other metal to obtain the desired conductivity for sparking the sample. Estimated accuracy of the spark source is a factor of three.

Neutron Activation

The copper (II) fluoride samples were activated in one of the Hanford production reactors, which are noteworthy for uniformity of the highly thermalized neutron flux. The activated samples were then counted by specialized methods including multi-dimensional gamma ray spectral analysis and anti-coincidence shielded solid state detector (lithium-drifted germanium diodes) techniques. The solid state detector systems provide very high resolution, and, when used in anti-coincidence systems, take advantage of a sharply reduced background. The estimated accuracy of neutron activation is $\pm 30\%$. The technique can be used for analysis of Co, Sb, Sc, Zn, Ag, Cs, U, Se, Cr, Fe, Hg, Na, Mn, Br, Ni, Rb, W, Ca, Hf, Ir, Cd, In, As and several rare earths in copper (II) fluoride.

Emission Spectroscopy

A Bausch and Lomb "Varisource" emission spectrograph was used for analysis. The estimated accuracy is a factor of two.

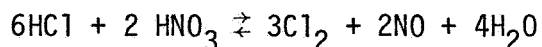
METHODS OF PREPARING COPPER (II) FLUORIDE

There are many possible experimental techniques which one might investigate to prepare ultra pure copper (II) fluoride. In this program we studied in detail the following three methods: 1) Fluorination of copper (II) sulfide; 2) Decomposition of a copper (II) ammonium fluoride double salt;^(1,2) and 3) Fluorination of copper (II) hydroxyfluoride.

Fluorination of Copper (II) Sulfide

High purity copper metal (99.999%) was dissolved in nitric acid. After dissolution of the metal, the nitric acid was removed by addition of

concentrated hydrochloric acid followed by boiling. The reaction is shown below:



This step is necessary to prevent the oxidation of hydrogen sulfide to sulfur when adding hydrogen sulfide to the solution. Some of the excess hydrochloric acid is removed by boiling down the copper (II) chloride solution to a level where copper compounds do not quite precipitate out. The copper (II) chloride solution is then diluted with water and hydrogen sulfide is added to precipitate copper (II) sulfide. The copper (II) sulfide is centrifuged six times, with the material rinsed thoroughly with water between each step. The copper (II) sulfide is then dried at 120°C in flowing air, ground to a fine powder in a copper mortar and pestle and then fluorinated according to the following reaction: $\text{CuS}_{(\text{c})} + 4\text{F}_{2(\text{g})} \rightarrow \text{SF}_{6(\text{g})}$. The main difficulty with this technique was that the reaction was so vigorous "clinkers" would form during the fluorination step. These clinkers would form even when the fluorine was diluted with nitrogen. Clinker formation caused local incomplete fluorination, so that grinding between each fluorination step was required to obtain ultra pure copper (II) fluoride free of sulfide. The experimental conditions involved passing 50-80% fluorine over the copper (II) sulfide at 100 to 200 cc/min. The temperatures ranged from 275°C to 375°C. Some typical analyses of the copper (II) sulfide and the resulting copper (II) fluoride are shown in Table I.

It is fairly easy to prepare copper (II) sulfide which is low in impurities. In the copper (II) sulfide sample of Table I only sodium and iron are approaching the maximum impurity level which could cause problems in the final product. Extended fluorination at 500°C should lower the concentration of chloride, oxygen, sulfur and carbon to acceptable levels. It is obvious from the copper (II) fluoride analysis that a single fluorination is inadequate. Also, some iron, nickel and chromium were apparently picked up during the fluorination step. This impurity pickup would have to be minimized before this technique could be used for the preparation of ultra pure copper (II) fluoride in kilogram quantities. Since the copper (II) sulfide was readily passivated in a fluorine atmosphere, it is difficult to drive this reaction to completion. For this reason, the technique was not studied any further.

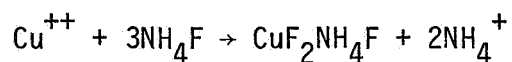
TABLE I

SPARK SOURCE MASS SPECTROMETER ANALYSIS OF COPPER (II) SULFIDE
AND COPPER (II) FLUORIDE

<u>Impurity</u>	<u>CuS</u>	<u>CuF₂</u>
Ag	4	< 2
Zr	4	< 2
As	2	6
Ni	5	55
Fe	35	300
Mn	3	15
Cr	10	50
Ca	8	20
K	15	10
Cl	30,000	1,500
S	---	20,000
P	2	8
Si	< 45	< 15
Al	< 2	15
Mg	25	30
Na	45	30
O	6,000	4,000
N	15	---
C	700	1,000

Decomposition of a Copper (II) Ammonium Fluoride Double Salt

High purity copper metal was again dissolved in nitric acid. The concentrated acidic copper nitrate solution was then added to a saturated solution of ammonium fluoride. This reaction is shown below:



The resulting copper (II) ammonium fluoride double salt was washed six times with a saturated ammonium fluoride solution and centrifuged between each washing operation. About half of the original double salt is lost in this washing process. The wet double salt was placed on a pure copper sheet and dried under flowing air at 120°C. We attempted to decompose the double salt under hydrogen fluoride at elevated temperatures but were unsuccessful, as it was impossible to remove all of the water from the double salt without forming a copper oxide or copper oxyfluoride intermediate. This problem was solved by decomposing the double salt under fluorine at 500°C. To minimize the amount of ammonium fluoride condensing out in the exit lines the double salt was heated to 250-350°C under argon to remove most of the ammonium fluoride prior to fluorination.

This technique worked quite well for preparing pure copper (II) fluoride. Analyses of the copper (II) fluoride prepared from the double salt are shown in Table II.

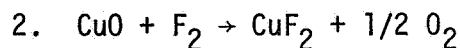
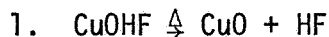
TABLE II

SPARK SOURCE MASS SPECTROMETER ANALYSIS OF COPPER (II) FLUORIDE
PREPARED FROM COPPER (II) AMMONIUM FLUORIDE DOUBLE SALT

<u>Impurity</u>	<u>First Fluorination</u> <u>CuF₂</u>	<u>Second Fluorination</u> <u>CuF₂</u>
Fe	20	
Ti	< 1	
Ca	4	
K	50	
Si	< 20	
Al	4	
Mg	30	
Na	20	
C	200	4 ppm
Cl	20	
O	2,000	9 ppm
Ni	10	
Mn	17	
Cr	20	
N		< 1 ppm

Fluorination of Copper (II) Hydroxyfluoride

Ultra pure copper (II) hydroxyfluoride makes an excellent starting material, as it can be purchased on the open market containing less than 10 ppm of any metallic impurity. Copper (II) fluoride can be easily made by directly fluorinating the copper (II) hydroxyfluoride according to the following reactions.



This compound is not appreciably passivated by fluorine and the reaction goes to completion. In reaction #1 finely divided and active copper oxide is formed which reacts with the fluorine as it is formed, tending to inhibit the formation of clinkers. Furthermore, the liberation of hydrogen fluoride further tends to break up clinkers. The decomposition products from reaction #1 and #2 are gaseous and easily removed; consequently, a pure copper (II) fluoride product is left. A typical analysis of the starting copper (II) hydroxyfluoride material and the resulting copper (II) fluoride are shown below in Table III.

TABLE III

SPARK SOURCE MASS SPECTROMETER ANALYSIS OF COPPER (II)
HYDROXYFLUORIDE AND COPPER (II) FLUORIDE

	<u>CuOHF</u>	<u>CuF₂</u>
Fe	.6	35
Ti	.9	--
Ca	4	40
K	5	25
Si	< 20	20
Al	< 2	15
Mg	< 10	15
Na	.3	40
C	4,000	50
Cl	10	10
O	---	80
Ni	N.D.	15
Mn	N.D.	30
Cr	N.D.	15
P	N.D.	20

In the initial experiments there were some impurities picked up during the fluorination step and while handling the material. In subsequent experiments the impurity pick-up was nearly eliminated and the oxygen concentration was lowered below 50 ppm. The fluorination of copper (II) hydroxyfluoride was chosen as the route to take for the preparation of one kilogram of ultra pure copper (II) fluoride.

PREPARATION OF ONE KILOGRAM OF ULTRA PURE
COPPER (II) FLUORIDE

To assure that the final kilogram of copper (II) fluoride prepared by fluorinating copper (II) hydroxyfluoride would meet purity specifications, it was necessary to generate confidence in the analytical procedures used in the final assessment of product purity. NASA Lewis submitted a standard copper (II) fluoride sample to Battelle-Northwest for analysis. The standard sample was analyzed by spark source mass spectroscopy, neutron activation and emission spectroscopy. These results were compared with values obtained from NASA; the data as determined by another laboratory is shown in Table IV. The general agreement between the various methods is good except for zinc and tin, where cross-contamination may be responsible for high results. Since the Battelle-Northwest spark source agreed reasonably well with the other techniques of analysis, this method was relied on to follow the impurity level of the copper (II) fluoride product.

Ultra pure copper (II) hydroxyfluoride was purchased from the Ledoux Company. Spark source analyses of two one-thousand gram lots are shown in Table V. The carbon and chloride values are quite high, but, as mentioned before, they can be lowered to acceptable levels during the fluorination process.

TABLE IV
ANALYSIS OF COPPER (II) FLUORIDE STANDARD

<u>Impurity</u>	<u>NASA</u>		<u>Battelle-Northwest</u>		
	<u>Spark Source</u>	<u>Emission Spectroscopy</u>	<u>Spark Source</u>	<u>Neutron Activation</u>	<u>Emission Spectroscopy</u>
H	27				
Li	.1				
B	10.2				200
C	114		30		
N	31				
O	1230		3700		
Na	204		17		
Mg	42	33	19		200
Al	104	170	62		< 100
P	11		2		
S	340		74		
Cl	260		7		
K	25		9		
Ca	21	86	92		
Ti	<10		5		
V	45		1		
Cr	13	5	1	6.4	
Mn	12	23	1		
Fe	1200	1100	460	1000	1000
Ni	730	970	520		1000
Zn	250#		18	80	
Ga	2.7				
As	<11		6		
Ag	26	6	5	9.5	
Cd	70				
Sn	4600	400	49	180	200
Te	38				
Pb	270	120	3		200
Bi	300				
Si			230		50

#May be due to cross-contamination

TABLE V

SPARK SOURCE ANALYSIS OF COPPER (II) HYDROXYFLUORIDE

<u>Impurity</u>	<u>CuOHF Lot #2</u>	<u>CuOHF Lot #3</u>
Fe	4	.6
Ti	4	6
Ca	2	1
K	20	4
Si	< 40	< 6
Al	< 2	9
Mg	50	30
Na	10	7
C	1200	1000
Cl	100	50

The copper (II) hydroxyfluoride was fluorinated in two steps because it contained a significant amount of water. The first fluorination dehydrated the copper (II) hydroxyfluoride completely and left pure copper (II) fluoride containing about 1000 ppm by weight oxygen. The fluorination could not be carried out in one step as the presence of water in the reaction vessel caused corrosion of the copper liner and produced a non-adherent film on the copper shelves used to contain the copper (II) hydroxyfluoride. Thus extended fluorinations would contaminate the copper (II) fluoride with incompletely fluorinated copper metal. The following procedure was used to prefluorinate one kilogram of ultra pure copper (II) fluoride.

1. Load the reactor with 140 grams of copper (II) hydroxyfluoride, with the material divided equally among 3 copper shelves.
2. Dry overnight at 170°C with a nitrogen flow of 80 cc/min.
3. Passivate the reactor at 170°C with a fluorine flow of 100 cc/min and a nitrogen flow of 100 cc/min for 10 minutes. Then passivate again at a fluorine flow of 200 cc/min and a nitrogen flow of 30 cc/min for 10 minutes.
4. Elevate the temperature to 400°C and hold at that temperature for 5-6 hours, keeping the fluorine flow at 200 cc/min and the nitrogen flow at 80 cc/min.
5. Cool, purge reactor with nitrogen and store the copper (II) fluoride in a dry box to prevent any unnecessary pick up of moisture.

For the final fluorination and packaging steps the following procedure was used.

1. Grind 120 grams of copper (II) fluoride with a copper mortar and pestle and place in the reaction vessel, dividing the material equally among 3 shelves.
2. Passivate the reactor at 170°C with a fluorine flow of 100 cc/min and a nitrogen flow of 100 cc/min for 10 minutes. Then passivate again with a fluorine flow of 200 cc/min and a nitrogen flow of 30 cc/min for 10 minutes.
3. Elevate the temperature to 480°C with a fluorine flow of 200 cc/min and a nitrogen flow of 80 cc/min. Hold the temperature at 480°C for 5 to 6 hours.
4. Cool to room temperature, then sparge out the fluorine with nitrogen. Place the sealed reactor into a dry box.
5. Analyze the copper (II) fluoride for oxygen by spark source mass spectroscopy. If the oxygen concentration is too high repeat Steps 1, 2, 3, 4, and 5.
6. The quartz bottle and adapter (shown in Figure III) is evacuated to approximately 1×10^{-5} mm Hg.
7. The quartz bottle is heated to about 500°C to drive off any adsorbed water. The quartz neck is heated as high as possible without damaging the viton O-ring.
8. The greaseless stopcock is turned off and the evacuated system is placed in a nitrogen atmosphere dry box and vented to one atmosphere.
9. Remove the adapter and quartz plug.
10. Insert a copper shield into the neck of the bottle, then insert the long stem copper funnel. This arrangement minimizes the amount of copper (II) fluoride adhering to the quartz neck when the funnel is pulled out.
11. Place the quartz plug in the neck and attach the adapter with the stopcock turned off.

12. Attach the assembly to a vacuum rack.
13. The capsule is partially evacuated and the quartz neck is fused and terminated at the quartz plug.

Each of the 100 gram lots of copper (II) fluoride prepared in the above manner were analyzed for oxygen to assure that the reaction had gone to completion. These analyses are shown in Table VI, including a typical analysis for hydrogen. The metal impurities were estimated by preparing a composite copper (II) fluoride sample made up from equal quantities of each of the ten copper (II) fluoride lots. Spark source analysis of the composite sample is shown in Table VII. Analysis of the composite sample by emission spectroscopy is shown in Table VIII.

In general, the analysis from the spark source agreed well with the emission spectrograph, although 75 ppm magnesium was found using the emission spectrograph. Even including this high magnesium value the final copper (II) fluoride product is still 99.98% pure.

TABLE VI
OXYGEN AND HYDROGEN ANALYSIS BY SPARK SOURCE

<u>Sample Number</u>	<u>CuF₂ Lot Number</u>	<u>Oxygen (ppm)</u>	<u>Hydrogen (ppm)</u>
F-3-BA	1	70	--
F-3-BD	2	40	4
F-3-BE	3	30	--
F-3-AF	4	60	--
F-2-BG	5	10	--
F-2-AH	6	20	--
F-2-BI	7	7	--
F-2-AJ	8	60	--
F-2-BK1	9	20	--
F-2-BK2	10	20	--

Average oxygen concentration: 34 ppm

TABLE VIIANALYSIS OF COPPER (II) FLUORIDE COMPOSITE
BY SPARK SOURCE

<u>Impurity</u>	<u>Analysis #1 ppm</u>	<u>Analysis #2 ppm</u>	<u>Average 1 & 2 ppm</u>
Co	2 ppm	6 ppm	4 ppm
Ni	2	1	1.5
Fe	6	6	6
Cr	0.5	0.5	0.5
Ca	4	10	7
K	10	4	7
Na	20	8	14
N	1	1	1
C	20	10	15
O	--	--	34#
H	--	--	4##

Estimate of total impurities by spark source: 94 ppm

Average oxygen concentration from Table VI

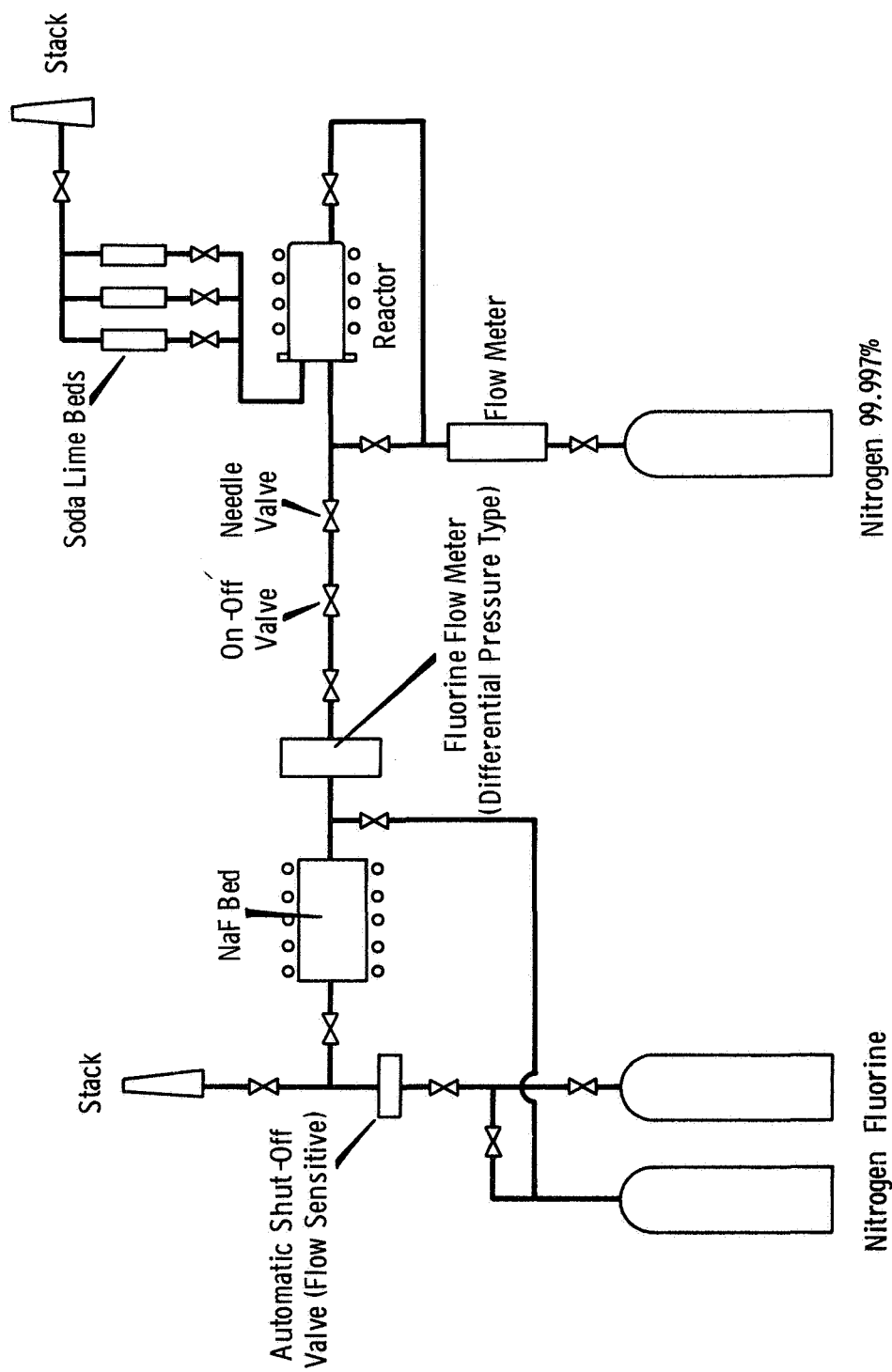
A single hydrogen determination

Spark source mass spectrography analyses are estimated to be correct within a factor of 3. The analyses are reported in ppm by weight. Elements not listed are below 10 ppm except for copper and fluorine.

TABLE VIIIANALYSIS OF COPPER (II) FLUORIDE COMPOSITE
BY EMISSION SPECTROSCOPY

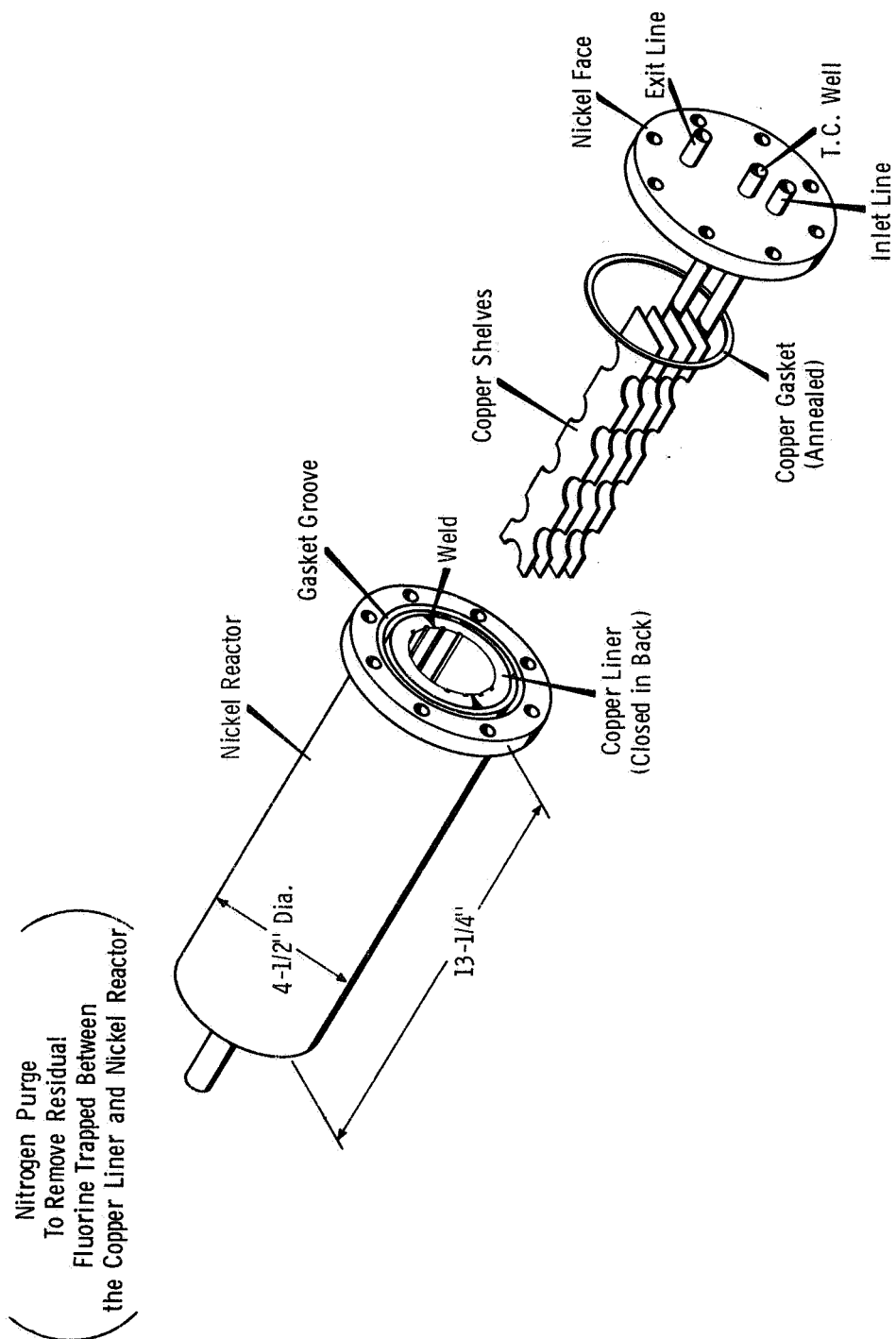
<u>Impurity</u>	<u>ppm</u>	<u>Impurity</u>	<u>ppm</u>
Ag	ND	Mo	ND
Al	ND	Na	ND
As	< 50	Nb	ND
B	ND	Ni	ND
Be	ND	P	ND
Bi	ND	Pb	< 20
Ca	< 20	Pd	ND
Cd	ND	Pt	ND
Co	ND	Rh	ND
Cr	ND	Ru	ND
Fe	< 100	Sb	ND
Ga	< 20	Si	< 50
HF	ND	Sn	ND
In	ND	Ta	ND
Ir	ND	Ti	ND
K	ND	V	ND
Li	ND	Y	ND
Mg	75	Zn	ND
Mn	ND	Zr	ND

Emission spectroscopy analyses are estimated to be reliable within a factor of 2.



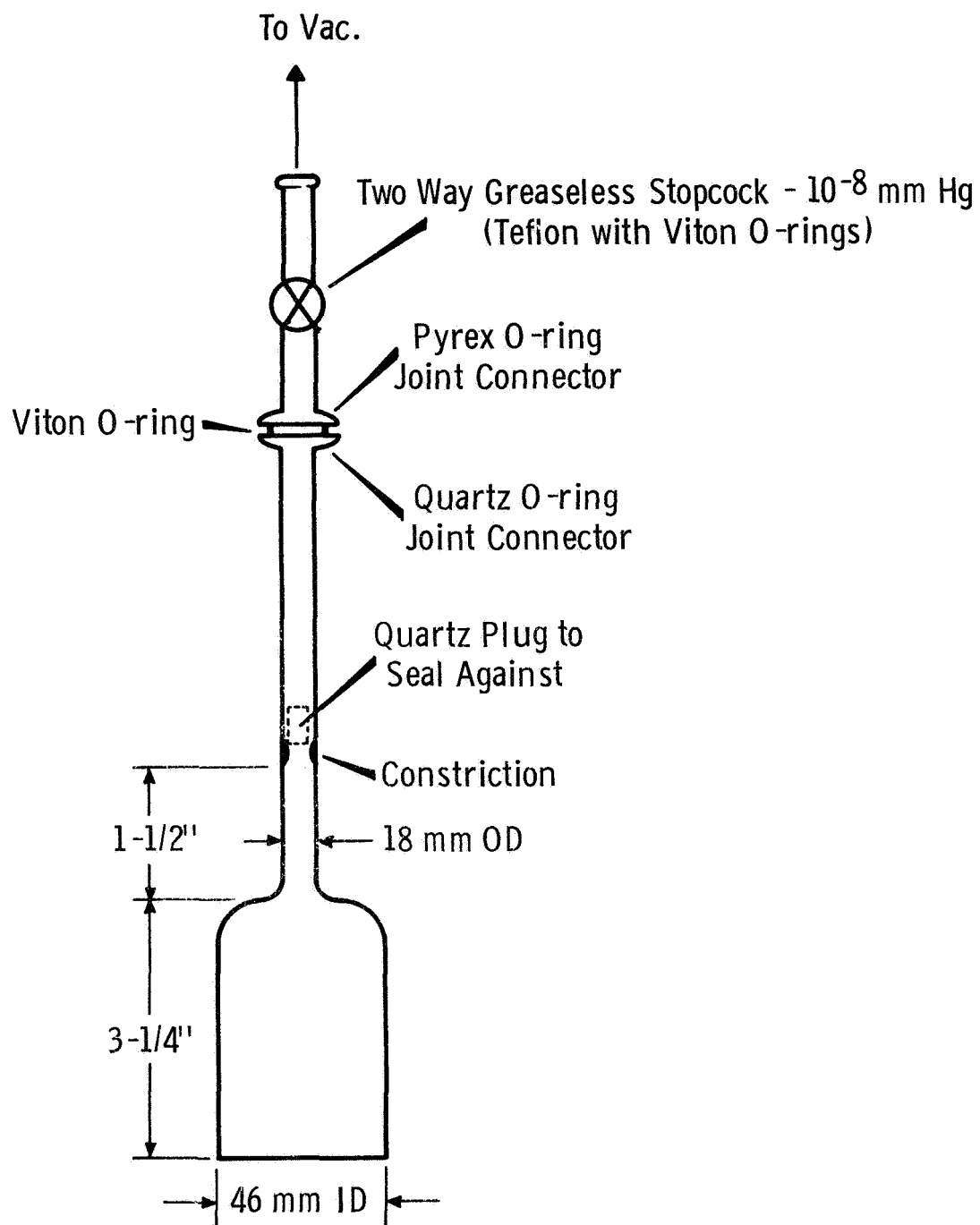
Fluorine Facility

FIGURE I



Reaction Vessel

FIGURE II



Quartz Bottle and Adapter

FIGURE III

REFERENCES

1. H. M. Haendler, et. al., "The Synthesis of Ammonium Fluorometallates in Methanol", J. Am. Chem. Soc., 80, 2662 (1958).
2. H. M. Haendler, et. al., "The Synthesis of Ammonium Fluorometallates in Methanol", J. Am. Chem. Soc., 82, 4158 (1960).